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REMARKS

The applicants note with appreciation the acknowledgement of the claim for priority under section 119 and the notice that all of the certified copies of the priority documents have been received.

The applicants acknowledge and appreciate receiving a copy of form PTO-1449, on which the examiner has initialed both listed items.

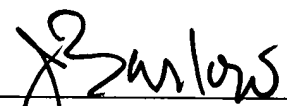
Claims 1-10 are pending. The applicants respectfully request reconsideration and allowance of this application in view of the above amendments and the following remarks.

Claims 1-10 were rejected under 35 USC 112, second paragraph, as being indefinite. The claims have been clarified in the amendments above and are now more readable. In addition, copies of three technical articles are enclosed to show that the applicants are using standard terminology and nomenclature to designate the crystallographic planes. Therefore, the applicants respectfully request examination of the claims on the merits.

If questions relating to patentability remain, the examiner is invited to contact the undersigned by telephone.

Please charge any unforeseen fees that may be due to Deposit Account No. 50-1147.

Respectfully submitted,



James E. Barlow
Reg. No. 32,377

Posz & Bethards, PLC
11250 Roger Bacon Drive, Suite 10
Reston, VA 20190
Phone 703-707-9110
Fax 703-707-9112
Customer No. 23400

Slicing Off-Axis Si, SiGe, and Ge Wafers

Stephen H. Jones, Michael H. Jones

August 2002

Virginia Semiconductor, Inc.

1501 Powhatan Street, Fredericksburg, VA 22401

(540) 373-2900, FAX (540) 371-0371

www.virginiasemi.com, shjones@virginiasemi.com

A. Introduction

Many engineers and device designers that are fabricating microelectronic, photonic, and Microelectromechanical devices and circuits require silicon wafers cut off-axis to a primary surface. Surfaces vicinal to the $\{100\}$, $\{111\}$, and $\{110\}$ are most common and produced by cutting an on-axis crystal at the appropriate angle. Virginia Semiconductor Incorporated (VSI) can grow crystals of the $\{111\}$, $\{100\}$, $\{110\}$, $\{211\}$, $\{311\}$ crystallographic direction. Also, VSI can slice the ingot along any axis to produce wafers with surfaces at any position on the stereographic projection. This paper describes the basic process and descriptions used to cut off-axis wafers.

The typical Integrated Circuit (IC) industry uses off-axis $\{111\}$ wafers for certain applications in device and circuit fabrication. There exist SEMI Standards for this classification of $\{111\}$ wafers, and the related, general, SEMI diagrams for these wafers are given below (Figure 1). This paper, and VSI customers in general, are not as concerned with specifics of the off-axis $\{111\}$ wafer, but various specification wafers that are off-axis to any primary surface. One should refer to SEMI M1-0298 if the details of off-axis $\{111\}$ wafers are of interest. A general description of the Miller $\{hkl\}$ planes and Miller $\langle hkl \rangle$ directions is given in reference [1].

Seeded PVT Growth of Aluminum Nitride on Silicon Carbide

B.M. Epelbaum^{1,2}, M. Bickermann¹ and A. Winnacker¹

¹Department of Materials Science 6, University of Erlangen-Nürnberg,
Martensstr. 7, D-91058 Erlangen

²SiCrystal AG, Paul-Gossen-Str. 100, D-91052 Erlangen

Keywords: aluminum nitride, AlN seeded growth, SiC seeds for AlN,
SiC substrate orientation, a-plane growth

Abstract. Seeded bulk crystal growth of AlN was conducted by physical vapor transport (PVT) using AlN powder as a starting material. SiC substrates with different crystallographic orientation were used as seeds. Materials compatibility was investigated; temperatures higher than 2000°C lead to decomposition of the SiC seed and to degradation of the tungsten heaters. Hexagonal hillocks grow on c-plane SiC seeds leading to growth instabilities and layer quality deterioration, whereas for growth on a-plane seeds millimeter-sized a-oriented areas were obtained showing a smooth morphology typical for step-flow growth mode. A comparison of the natural growth habits of freely nucleated SiC platelets and prismatic elongated AlN can explain this behavior. The best crystal quality was obtained by employing slightly off-oriented a-plane SiC wafers at growth temperatures below 2000°C, but growth rate and surface diffusion necessary for step-flow growth remain insufficient.

Introduction

Bulk crystals of AlN have a great potential to provide a better substrate for group-III nitride electronic devices than currently available sapphire and silicon carbide [1]. Despite a number of promising crystal growth studies on AlN [3-5] published after the pioneering work of Slack and McNelly [2], available crystals are still too small to allow their further use in seeded growth. Very recently Schlessner, Dalmau and Sitar demonstrated the feasibility of PVT seeded growth on small-sized spontaneously nucleated AlN platelets [6], but for production of larger crystals silicon carbide remains to be the only readily available choice for seeding. Balkas et al. [3] and Sarney et al. [8] grew AlN layers on on-axis 6H-SiC, but deposited layers contain multiple grains of millimeter size. Finally, Liu et al. have achieved an improvement of the AlN layer quality by applying buffer layers of MOCVD grown AlN prior to vapor growth. One critical shortage of SiC seeds in AlN sublimation growth is the problem of poor chemical compatibility of these materials as emphasized in [7]. In our present experimental study we have investigated the dependence of the AlN layer surface morphology and crystal quality on the SiC substrate orientation.

Experimental procedure

Crystal growth experiments were conducted in a small resistively heated growth reactor provided with tungsten heating elements capable up to 2500°C in the atmosphere of high-purity nitrogen, which was described in details in our previous work [7]. Most of the results discussed below were obtained at temperatures around 2000°C. A flow of 5N purity nitrogen through the reactor volume at 50-70 sccm was established, while the total pressure was kept at 350 mbar, i.e. nearly stagnant gas flow conditions were applied. The source material was AlN powder with 99.5% purity (main residual impurity is oxygen) supplied by H.C. Starck GmbH. Seed plates 10x10 mm² in size and having different orientations were cut from 6H-SiC crystals grown in our laboratory and mounted inside a graphite holder coated by a protective SiC layer. The utilization of the

protective coating was essential to prevent unacceptable losses of the AlN charge. Alternatively, an AlN ceramic holder was used for growth experiments where seed temperature exceeded 2000°C. Typically, a growth time of two hours was sufficient to produce AlN layers with a thickness about 200-500 μm. The grown layers were characterized using scanning electron microscopy (SEM).

Results and discussion

Consideration of processing conditions and materials compatibility

The equilibrium pressures of gaseous components over solid AlN and SiC decisively determine the favorable conditions of seeded AlN growth. As can be seen in Fig. 1, processing conditions for pure SiC and AlN growth are strongly different. Growth of AlN requires much higher ambient (N₂) pressure and lower temperatures compared to SiC growth. This seemingly makes SiC quite a suitable seeding material. However, experimental verifications made previously [3,4] and supported by our recent results on materials compatibility in the Al-N-Si-C system [7] show that there exists a drastic difference in equilibrium pressures in the quaternary system Al-N-Si-C and the corresponding binary Al-N and Si-C systems. Noticeable growth of an AlN-SiC mixed crystal takes place already at 1800-1900°C, assisted presumably by the formation of volatile Al₂OC due to inevitable oxygen contamination of the starting AlN powder [2,9]. Growth rate of pure AlN in this temperature range is still very low. In order to obviate this difficulty, SiC material (seed and seed holder) was used exclusively in a cold end of our arrangement and any contamination of charge AlN powder with SiC was carefully avoided. A moderate seed temperature of about 2000°C was found to be appropriate with respect to the integrity of the tungsten heating elements. Hence, growth experiments were conducted at 2000°C and a nitrogen pressure of 350-400 mbar.

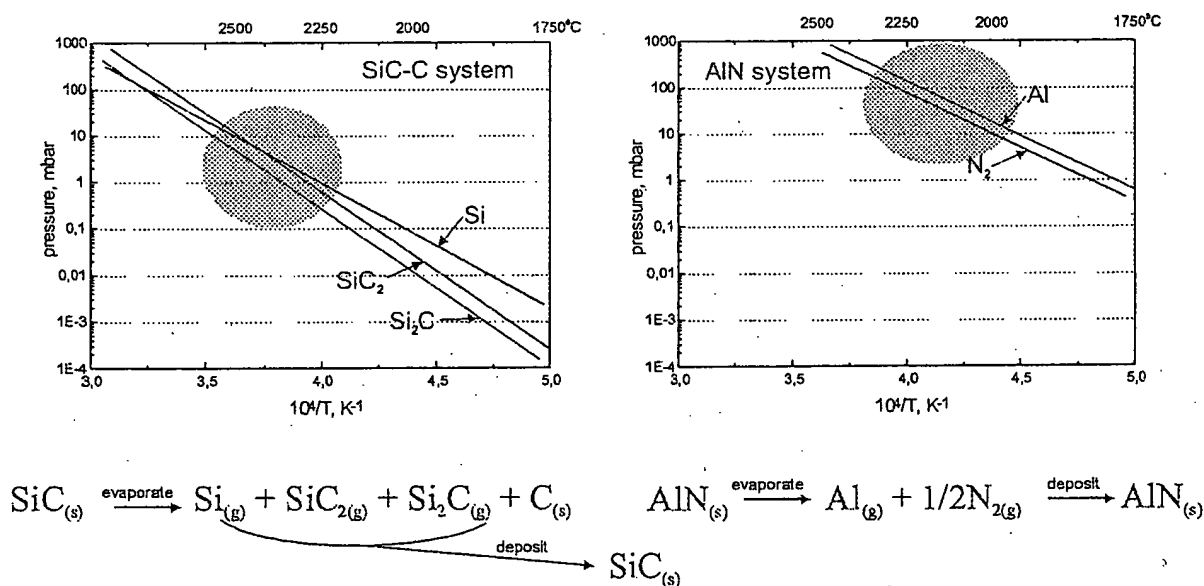


Fig. 1: Comparison of growth parameters for SiC and AlN single crystal growth by PVT. The gray field represents the domain of partial pressures and temperatures relevant for effective crystal growth by direct sublimation. Data were taken from [10,11].

Growth results

During growth on the Si face of c-oriented SiC substrates a large number of sharp hexagonal hillocks was formed covering the entire growth surface of the AlN layer, see A and B in Fig. 2. Although epitaxial growth in c-direction was easily achieved (note the nearly perfect hillock

alignment in Fig. 2 B), this morphology is unfavorable for the preparation of high-quality layers since it often leads to growth instabilities and even a transition to polycrystalline growth after the first 100-150 μm can be observed. The reason for this is the frequent formation of voids and subgrains at the places where pyramidal faces of two neighboring hillocks meet each other as shown in Fig. 3 A.

Growth on approximately 10° off-axis a-oriented 6H-SiC substrates was found to be more stable. In this case, the growth surface was also completely faceted and covered by elongated a-faces of AlN, see Fig. 2 C. The best results were achieved with on-axis a-oriented substrates, Fig. 2 D and Fig. 3 B, where millimeter-sized a-oriented areas were obtained showing a smooth morphology typical for step-flow growth mode. However, in the case of on-axis a-orientation we also observed a number of thermal etch pits appearing on the surface. This seems to be a particular feature of the early stage of gas phase bulk growth, since similar structures can also be found on SiC crystals, if the growth process is interrupted within the first hours. We made an attempt to increase the growth temperature by employing an AlN ceramic holder for the SiC seed. At 2100-2150°C, large a-plane faceted areas were grown within the same processing time of two hours, but unfortunately the tungsten heating elements were strongly damaged already after first growth run.

In almost all cases mechanical stresses introduced in SiC substrates after growth were large enough to cause cracking as shown in Fig. 2 B. The cracks sometimes force the AlN layer to peel off from SiC, and as they originate typically in the SiC wafer, a thin SiC layer is still attached to the grown AlN layer.

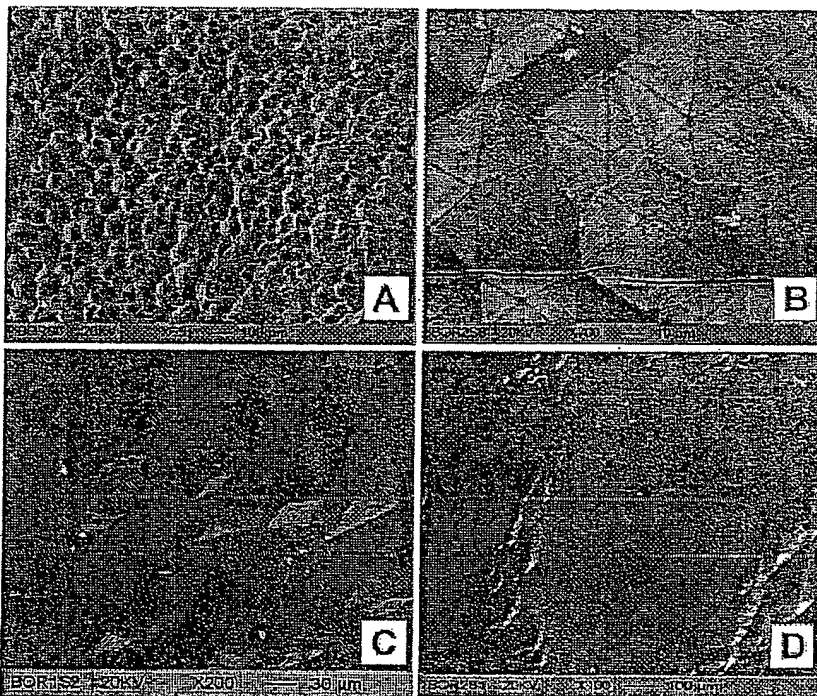


Fig. 2. Surface morphology of AlN layers grown on variously oriented SiC substrates: A and B – grown on Si face on-axis c-oriented 6H SiC, C – grown on 10° off-axis a-oriented 6H-SiC, D – grown on on-axis a-oriented 6H SiC.

Optimal choice of SiC substrate orientation

A comparison of the natural growth habits of freely nucleated Lely platelets of SiC [12], where the c-axis is the slowest growth direction, and prismatic elongated AlN [7] with the c-axis being the fastest growth direction can explain the problems of AlN seeded growth on standard (0001) 6H-SiC wafers. We have estimated the growth rate anisotropy in AlN grown at 2000°C and found that the growth rate along the c-axis is about 5 to 7 times higher than the growth rate in perpendicular growth directions. Formation of the c-plane facet on AlN in seeded growth is nearly

impossible in this case, since the quickly growing c-plane facet disappears and is substituted by multiple pyramidal faces, accompanied by layer quality deterioration. The best choice for the SiC substrate orientation to growth thick layers of AlN is to use slightly ($1-3^\circ$) off-oriented a-wafers. Note that this conclusion is relevant only for temperatures of about 1900-2000°C, since at higher temperatures the growth rate ratio may change. Furthermore, temperatures below 2000°C seem to be too low to enhance the surface diffusion of the constituent atoms and hence are insufficient for producing a step-flow mode necessary for effective bulk growth of AlN.

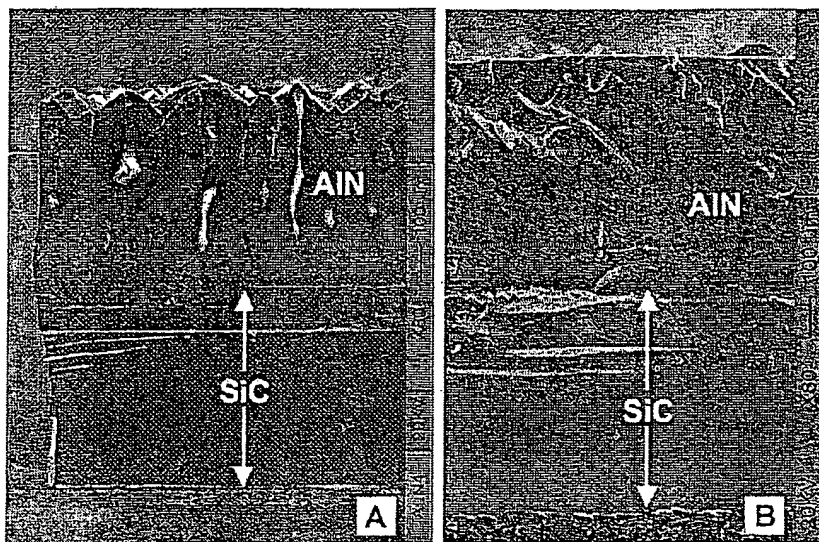


Fig. 3. SEM pictures of cleaved surfaces through the SiC substrate and PVT grown AlN layer. A corresponds to A and B in Fig.2, B corresponds to D in Fig.2.

Conclusions

The optimal choice for the substrate orientation to growth thick layers of AlN on SiC is to employ slightly off-oriented a-plane SiC wafers. An increase in growth temperature leads to larger areas with smooth morphology typical for step flow growth mode, but simultaneously decomposition of the SiC seed and formation of the mixed AlN-SiC compound is observed. Actually, the effective utilization of a SiC seed is limited to temperatures lower than about 2000°C.

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Homoepitaxial Growth of High-Quality 6H-SiC Thin Films by Using Chemical-Vapor Deposition with Bis-trimethylsilylmethane at a Low Temperature

Jae Kyeong JEONG, Hoon Joo NA, Yeon Shik CHOI and Hyeong Joon KIM*

Division of Materials Science and Engineering, Seoul National University, Seoul 151-742

Wook BAHNG

Materials Science Division, Electrotechnical Laboratory, Ibaraki 305, Japan

High-quality, monocrystalline 6H-SiC thin films were epitaxially grown on 6H-SiC (0001) substrates, which were 3.5° off-axis from [0001] toward [11 $\bar{2}$ 0], at 1320 °C by organo-metallic chemical-vapor deposition. We used bis-trimethylsilylmethane [$C_7H_2Si_2$], which is a non-toxic and non-flammable organosilicon source having an alternate SiC bond structure. Lateral growth of the SiC film was observed by scanning electron microscopy and atomic force microscopy. Triple-axis crystal diffraction analysis showed a strain-free epitaxial layer with a 11.4-arcsec full width at half maximum, whose quality was comparable to that of the substrate. The photoluminescence spectrum revealed that the 3C-SiC polytype was not included in the epitaxial layers.

I. INTRODUCTION

Silicon carbide (SiC) is an attractive semiconductor for high-temperature, high-frequency, and high-power devices due to its excellent electrical, thermal, and mechanical properties [1,2]. Also, homoepitaxial growth on a commercially available 6H-SiC wafer without lattice mismatch is promising rather than heteroepitaxial growth on silicon. Although high-quality 6H-SiC homoepitaxial films have been obtained using such methods as liquid-phase epitaxy (LPE) and vapor-phase epitaxy (VPE), there is the serious problem of the high growth temperature, which is above 1800 °C [3,4]. Recently, the growth temperature was dramatically reduced to 1500 °C by using slightly off-oriented 0001 6H-SiC substrate [5-6]. In chemical-vapor deposition (CVD) growth of SiC, the use of an off-orientation substrate makes it possible to duplicate the polytype of the substrate, as well as to grow laterally from atomic steps, hence leading to a significant improvement in terms of deposition temperature. However, a further reduction of growth temperature is still necessary to avoid the auto-doping problem and the redistribution of dopants. To solve this problem, we used an alternative organo-silicon precursor, which contained directly bonded Si and C atoms and decomposed at relatively low temperature. In addition, the organo-silicon precursor excluded the choice of the extremely pyrophoric SiH_4 from the process.

Although much research using various precursors has been done on the heteroepitaxy of 3C-SiC on silicon [7-10], there are no systematic reports for the homoepitaxy of 6H-SiC. In this paper, we report results on the growth of a 6H-SiC homoepitaxial film using a new class of organo-silicon source material, bis-trimethylsilylmethane [$C_7H_2Si_2$, BTMSM], which is neither toxic nor flammable.

II. EXPERIMENTAL PROCEDURES

6H-SiC substrates produced by Cree Research Inc. were used. The (0001) Si face of the substrate was 3.5° off-axis from [0001] toward [11 $\bar{2}$ 0], and the substrate was p-type with a net carrier concentration of $2.1 \times 10^{18} \text{ cm}^{-3}$. The epitaxial layer was deposited in a cold-wall, horizontal-type CVD reactor, where the substrates were heated using a graphite susceptor by radio-frequency induction heating. The typical substrate temperature was 1320 °C, which was measured by an optical pyrometer and calibrated using the melting temperature of Si (1418 °C). The experimental procedure has been presented in detail elsewhere [9]. The typical growth rate of the 6H-SiC thin films was 1.4 $\mu\text{m/hr}$. The thickness was estimated from Scanning electron microscopy (SEM) observations after the angle lapping of the 6H-SiC thin films by 5.4° .

The Laue back reflection method was used to determine the geometric off-axis direction. The surface morphology was observed by SEM and atomic force mi-

*E-mail: hjkim@plaza.snu.ac.kr, Tel: 02-880-7162, Fax: 02-884-1413

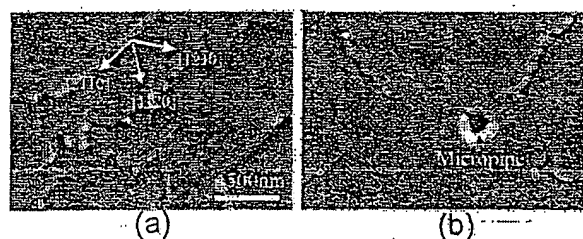


Fig. 1. (a) SEM image of the epitaxial layer with 1.4 μm thickness. The step configuration is clearly seen. The terrace width and step height measured by AFM are typically 1~2 μm and 10~60 nm, respectively. (b) The step feature around the micropipe is irregular. Note the micropipe penetrated the epitaxial layer.

croscopy (AFM). The polytype identification and the crystallinity of the epitaxial layer were analyzed by $\text{K}\alpha_1$ radiation by using a D3 X-ray diffractometer (Bede triple-axis crystal X-ray diffraction and photolumines-Scientific Instruments system), a Si (022) channel cut cence. Structural properties were characterized with Cuxrystal, and an analyzer. A computer-controlled goniometer with a precision of 0.055 arcseconds was used with high intensity beam geometry for a reciprocal space mapping of the off-axis (0006) plane. PL spectra were measured at 5 K by using a He-Cd laser with a wavelength of 3250 \AA a power of 30 mW.

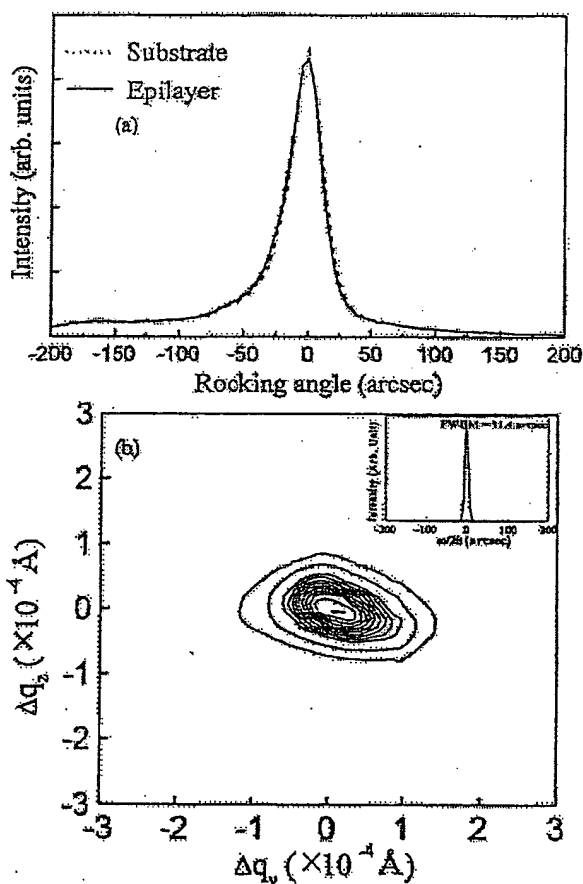


Fig. 2. (a) DCXRD of (0006) reciprocal lattice of 6H-SiC substrate and epilayer. (b) Reciprocal space mapping of (0006) plane of the epilayer. The directions of Q_y and Q_x are parallel to [1120] and [0001], respectively. The insert is a cross section through the map along the $\omega/2\theta$ direction.

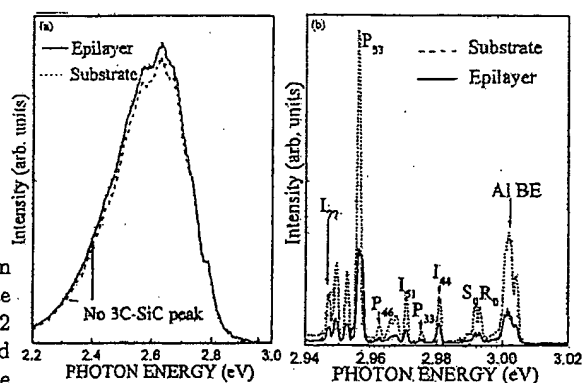


Fig. 3. PL spectrum of the substrate and the epitaxial layer (at 5 K, 30 mW).

III. RESULTS AND DISCUSSION

The surface of the as-grown layer is specular and smooth to the naked eye and optical microscope. Typical SEM images of an epilayer grown on the substrate (near 1320 $^{\circ}\text{C}$) are shown in Fig. 1. Macroscopic step bunching prevails through the entire area, which implies that lateral growth from the steps occurs. In other words, during the film growth the surface steps replicate 6H-SiC stacking sequence (ABCACB...), which is called the step controlled epitaxy [11]. But the epilayer has the hexagonal feature of the arrangement of regular macrosteps, which supports the atomistic step configuration model proposed by Davis *et al.* [12]. On the other hand, the micropipe, initiated from the micropipe on the substrate and developed through the whole thickness of the epilayer, seemed to disturb step propagation. Such a disturbance caused a wavy pattern near the micropipe, as shown in Fig. 1(b).

Table 1. Lattice constants of different SiC polytypes.

Polytype	a (\AA)	c/n (\AA)
3C ^a	3.083	2.517
6H ^b	3.091	2.530

^aLandolt-Bornstein: Numerical Data and Functional Relationships in Solid State Physics, Vol. 17, K.-H. Hellwege and O. Madelung (Springer-Verlag, Berlin, 1982).
^bA. Bauer, J. Krausslich, L. Dressler, P. Kuschnerus, J. Wolf, K. Goetz, P. Rev. B57, 2647 (1998).

In general, the narrow value of the full width at half maximum (FWHM) for an X-ray rocking curve indicates that the crystallinity of the material is excellent. The FWHM of a (0006) Bragg spot for single crystals is 31 arcsec and that of a high-quality epilayer is almost the same (32 arcsec), as shown in Fig. 2(a). If it is assumed that 3C-SiC grows on 6H-SiC in the manner of $(111)_{3C}/(0001)_{6H}$ [13], the (111) Bragg spot position of 3C-SiC is larger by $4.730 \times 10^{-4} \text{ \AA}^{-1}$ along the Q_z axis with respect to the (0006) Bragg spot position of 6H-SiC in reciprocal space. This value is obtained from the lattice constants of the 6H and 3C single crystals listed in Table 1. Considering the X-ray optic geometry under an ω -scan mode, the separation angle between $(111)_{3C}$ and $(0006)_{6H}$ in the fully relaxed state is 79 arcsec. However, the rocking curve shows only the peak of the 6H-SiC (0006) reciprocal point, which implies that 3C-SiC is not included. To analyze the broadening factors of FWHM, we characterized the epilayer by using the triple-axis crystal X-ray diffraction (TCD) method. Reciprocal mapping results of the (0006) Bragg spot show that the FWHM of the $\omega/2\theta$ scan is 11.4 arcsec. This validates the fact that the epitaxial layer is free from strain and has high quality. Also, the high ΔQ_y value in the reciprocal map reveals that the broadening of the double-crystal X-ray diffraction (DCXRD) rocking curve is caused by a mosaic spread rather than by lattice dilatation.

The photoluminescence spectra from the epitaxial layers using a graphite susceptor did not show the characteristic five-line near-band edge ($\sim 2.38 \text{ eV}$) [14] of the 3C-SiC polytype (Fig. 3(a)). This result is consistent with the result of the TCD analysis. In addition, when an Al acceptor recombines with a N donor on the hexagonal site and on the two quasi-cubic sites, the onsets of the acceptor-donor recombinations occur at 2.75 eV and 2.80 eV, respectively [15]. The slight increase in the donor-acceptor pair (DAP) transition band intensity and the minor decrease in the exciton peak intensity are ascribed to deep-level defects near the interface between the epilayer and the substrate, such as polishing scratches. The penetration depth for solids at wavelength λ is defined by $\delta_p = 1/\alpha = \lambda/4\pi k$, where α is the absorption coefficient and k the imaginary part of the complex index of refraction. Using the reported value of k [16] ($5.98 \times 10^{-3} \sim 7.65 \times 10^{-3}$ at 3.815 eV), the penetration depth at 325 nm can be estimated to be $4.32 \sim 3.38 \text{ \mu m}$. Because the film thickness is about 2 μm , the collected PL signal is comprised of that from the epilayer and that of the substrate. Consequently, it is believed that the broad DAP transition signal of the epilayer mainly comes not from the epilayer but from the substrate. The peak of the Al bound exciton is very clearly seen at 3.002 eV (Fig. 3(b)). Nitrogen bound exciton lines appear due to unintentional doping during the single-crystal growth [17]. Although the free and bound exciton peaks in the epilayer are reduced from those of the substrate, the existence of the free exciton peaks (I_{44} ,

I_{53} , and I_{77}) confirms that the epitaxial layer has high quality.

In general, as the off-axis angle of the substrate is increased, the deposition temperature to make homoeptaxy possible is reduced due to an increase in the step density [18]. To date, the lowest deposition temperature of a 6H-SiC homoeptilayer on a commercially available wafer with a 3.5° off-axis angle using CVD is 1450°C [6]. Our investigation shows the possibility of further reducing the deposition temperature if a bis-trimethylsilylmethane source is used. Further studies will focus on the PL analysis and the step morphology of a 10 μm -thick epilayer.

IV. CONCLUSIONS

We successfully deposited homoeptaxial thin films on 6H-SiC (0001) substrates which were 3.5° off-axis from $\langle 0001 \rangle$ toward $\langle 1120 \rangle$ at temperatures as low as 1320°C using a new type of organosilicon source, BTMSM. A lateral growth from steps made it possible to duplicate the substrate polytype at a low temperature. The FWHM of the (0006) plane for the substrate and the epilayer were 31 arcsec and 32 arcsec, respectively. The existence of the free exciton peaks (I_{44} , I_{53} , and I_{77}) in the photoluminescence spectrum confirms that the epitaxial layer has high quality.

ACKNOWLEDGMENTS

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